SPECIFICATIONS

Ammonium Glyphosate Synthesis Process

This invention concerns the preparation of glyphosate salts, in particular, the preparation of ammonium glyphosate.

Glyphosates are an effective, broad-spectrum, low-toxicity systemic, safe organic herbicide which is used after sprouting. Due to its excellent systemic propagation capability, it is very effective against many kinds of harmful weeds, and is widely utilized in agriculture, forestry, animal husbandry, gardening, etc. This herbicide is among those agricultural chemicals with the largest tonnage in the world.

Currently, China's domestic glyphosate herbicides contains 10% aqueous glyphosates, 41% aqueous isopropyl amine salt, 62% glyphosate isopropyl amine salts and 30% glyphosate soluble powder. As the use of glyphosate products expands both in China and throughout the world and their quality is continuously raised, competition grows stronger on a daily basis, and glyphosate compositions have been evolving into high-content, non –environmentally harmful compositions. When glyphosates are synthesized into ammonium glyphosate salt and processed into soluble powder products, they have a higher effect, the cost of their transportation and storage is reduced and they acquire the advantage of being easier to use, less harmful for the environment, etc.

The invention of Application No. 96196134.1 and CN 1192743A disclosed a method of preparing ammonium glyphosate by a reaction of glyphosate acid with a water solution of ammonium hydroxide (aqueous ammonia). However, since aqueous ammonia contains much water, this process is fairly complicated, the entire process of adding aqueous ammonia must be accompanied by continuous drying to maintain adequate moisture content, and the rate at which aqueous ammonia is added also must strictly controlled to ensure that the rate at which water is removed from the system exceeds the rate at which water enters the system; therefore samples must be taken at certain times to determine moisture content; moreover, the product also requires drying.

The invention of Application No. 96196133.3 and CN 1192742A disclosed a method of preparing ammonium glyphosate by a reaction of glyphosate acid with anhydrous ammonia gas. This method utilizes an "automated washing type" reactor and requires than "propeller" provided inside the reactor have precise dimensions to be able to continuously scrape off the product that is deposited on the reactor walls, otherwise, a solid lining would form on the walls, which could influence the reaction's heat transfer. It also requires a stringent control of the rate at which ammonia is added to make sure that the ammonia gas and the glyphosate acid enter the reactor in an entirely uniformly dispersed manner; if the ammonia input opening is faulty, it can easily become clogged with dirt and develop a conglomerate solid; the product must be roasted dry and pulverized.

In the application 99119971.5 submitted by these applicants, a reaction between glyphosate and liquid ammonia is used to prepare ammonium glyphosate but the reaction has to be completed in a pressure vessel.

The purpose of this invention is to overcome these deficiencies and offer a method of synthesizing ammonium glyphosate that has a straightforward technology,

stable product quality and a high yield, where the ammonium glyphosate synthesis would take place at relatively low pressure.

The characteristic features of this process of ammonium glyphosate synthesis involve the adding of an organic solvent to the reactor; after 2-8 hours of reaction between glyphosate and ammonia gas at 10 ~ 50 °C, the reaction liquid is cooled and the precipitated crystals are removed from it; the product resulting from the drying of crystals is ammonium glyphosate.

The mother liquid that remains upon the removal of crystals contains the solvent and a small amount of ammonium glyphosate; this mother liquid can be recycled to use as solvent in the next in the reaction of the next batch. At the same time a sufficient amount of solvent is added to replenish the lack of solvent due to partial loss of the mother liquid.

Said mother liquid can be recycled for use as solvent through a cycle set consisting of 6 ~ 15 batches. The final batch of mother liquid undergoes distillation and ammonium glyphosate is removed from it.

The solvent mentioned in this invention is methanol, ethanol, mineral ether, benzene, xylene or cyclohexane.

The optimal reaction temperature in this invention is 20~40°, the reaction time is 2.5 ~ 4 hours.

After this reaction, the reaction liquid can be cooled down to 15 ~ 20°C and the crystals removed from it.

The removed crystals can be can be dried by using a palletized drying oven, a revolving double cone dryer or fluidized layer dryer, and the solvent thus removed can be reused upon cooling and conversation recovery.

Since in this invention, ammonium glyphosate is synthesized by a reaction that glyphosate and ammonia gas in an organic solvent, the reaction proceeds at normal temperature and at normal pressure or a pressure of 0 – 0.5 MPa, the reaction process is straightforward and easy to operate and the cost of operation is low. Experiments and calculations demonstrate that the yield of the product of this invention can be as high as 97% or more; at the same time, this invention is advantageous in the case of product separation and drying.

Below we will explain the synthesis process of this invention referring to specific Practical Examples.

Practical Example 1. 170 g glyphosate original powder (95% purity) were placed in a 4-necked flask and into it was poured 500 ml methanol and ammonia gas whose number of moles is about 2-3 times that of glyphosate is passed through under stirring. The reaction temperature is controlled at about 25°C and after 3 hours of the stirring reaction, the reaction liquid is cooled to 20°C, and the precipitated crystals are filtered out. The crystals are baked dry, yielding ammonium glyphosate.

Practical Example 2. The experimental process and equipment are the same as in the Practical Example 1, except that 100 g water is added to the system prior to the reaction.

Practical Example 3. The experimental process and equipment are the same as in the Practical Example 1, except that industrial grade ethanol was used instead of methanol as a solvent.

Practical Example 4. The experimental process and equipment are the same as in the Practical Example 1, except that mineral ether (boiling point at 90°C) was used instead of methanol as a solvent.

Practical Example 5. The experimental process and equipment are the same as in the Practical Example 1, except that benzene was used instead of methanol as a solvent.

Practical Example 6. The experimental process and equipment are the same as in the Practical Example 1, except that xylene was used instead of methanol as a solvent.

Practical Example 7. The experimental process and equipment are the same as in the Practical Example 1, except that cyclohexane was used instead of methanol as a solvent.

Practical Example 8. Based on the Practical Example 1, the recovered mother liquid was used as solvent and recycled to carry out subsequent batch reactions.

Practical Example 9. The process and equipment are the same as in the Practical Example 8 except that the mother liquid was recycled and used no less than 5 times, and whenever it was used, methanol was replenished such that the amount of solvent reached 500 ml.

The table below gives the purity of the above product (in weight percent) and yield; the yield is calculated based on glyphosate (purity 95%)

Experiment	Ammonium glyphosate	Yield (%)
1	94.65	98.0
2	94.76	97.8
3	94.54	97.5
4	94.76	98.0
5	95.0	98.5
6	94.43	98.3
7	94.62	98.1
8	94.62	97.9
9	94.55	98.1

Practical Example 10. The process and equipment are the same as in either of the Examples 1 through 9, the reaction temperature is 10°; the reaction time is 10 hours.

Practical Example 11. The process and equipment are the same as in either of the Examples 1 through 9, the reaction temperature is 15°; the reaction time is 8 hours.

Practical Example 12. The process and equipment are the same as in either of the Examples 1 through 9, the reaction temperature is 20°; the reaction time is 5 hours.

Practical Example 13. The process and equipment are the same as in either of the Examples 1 through 9, the reaction temperature is 30°; the reaction time is 3 hours.

Practical Example 14. The process and equipment are the same as in either of the Examples 1 through 9, the reaction temperature is 35°; the reaction time is 2.5 hours.

Practical Example 15. The process and equipment are the same as in either of the Examples 1 through 9, the reaction temperature is 40°; the reaction time is 2.5 hours.

Practical Example 16. The process and equipment are the same as in either of the Examples 1 through 9, the reaction temperature is 50°; the reaction time is 2 hours.

Practical Example 17. The process and equipment are the same as in either of the Examples 1 through 9, prior to the reaction, 50 g water was added to the system; the reaction temperature is any temperature between 20 and 40°C; the reaction time is any time between 2 and 8 hours.

The measurements show that the product of this invention possesses stable quality and the purity and yield of the Examples 1 through 9 are basically the same.

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